



## ATTACHMENT A.2

### LIBBY OU3 PHASE I QUALITY CONTROL SAMPLE SUMMARY

#### ASBESTOS IN MINE WASTE

##### Data Completeness

Results have been reported for all of the field samples as specified in the Phase I SAP (USEPA 2007). The minimum requirements were met for field QC samples and laboratory QC samples. Note that the minimum number of laboratory QC samples as specified in the Phase I SAP (USEPA 2007) required for analysis is based on a minimum percentage across all media types.

##### Data Validation

In accord with the Phase I SAP, asbestos data were validated by selection of 10% of the samples at random and performing a detailed comparison of the FSDS and the laboratory bench sheets to the data recorded in the EDDs. A list of any omissions or apparent errors were submitted to the field team in the case of the FSDS forms and to the analytical laboratory in regard to analytical results reported in the EDDs. These issues were addressed and corrected. All tables and figures generated for this report reflect corrected data.

##### Field Quality Control Samples

**Field Duplicates.** A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision. Field duplicate results are ranked as concordant if both the original sample result and the field duplicate result report the same semi-quantitative classification. Results are ranked as weakly discordant if the original sample result and the field duplicate result differed by one semi-quantitative classification (e.g., Bin A vs. Bin B1). Results are ranked as strongly discordant if the original sample result and the field duplicate result differed by more than one semi-quantitative classification (e.g., Bin A vs. Bin B2). A total of 4 (11%) field duplicates were collected as part of the Phase I sampling program. All 4 field duplicates were analyzed by both PLM-VE and gravimetric analysis. As shown in Table 1, three of the original-duplicate pairs analyzed were in concordance and the other field duplicate was ranked as weakly discordant with the parent sample. This discordance may be due to analytical variability, but might also arise from authentic heterogeneity between the sediment samples. Because only four mine waste field duplicates were collected as part of the Phase I sampling program, the number of samples is too limited to draw firm conclusions regarding reproducibility. However, the data suggest that results will

generally be similar although differences due to small scale heterogeneity in the samples may occur.

**Performance Evaluation Samples.** Performance Evaluation (PE) samples are samples of a matrix that contain a known and certified level of a contaminant. The results of PE sample analysis help evaluate analytical accuracy. PE samples for LA in soil are available from USGS. These PE samples were prepared by mixing uncontaminated soil samples from Libby with known amounts of LA collected from the mine, so the true mass fraction of LA is known. A total of 19 PE samples with LA levels spanning a wide nominal range were evaluated at the PLM analytical laboratory prior to analysis of the Phase I samples as part of the laboratory mentoring and training program for Libby to evaluate laboratory proficiency. As described above for field duplicates, PE samples are ranked as concordant if the PE sample results report the same semi-quantitative classification as the nominal asbestos concentrations. As shown in Table 2, 13 (68%) of the PE samples were in concordance with the nominal values. Six of the PE samples were ranked as weakly discordant with the nominal values. Consistent with Libby Laboratory Modification #73 (LB-000073), proficiency is ranked as “good” if the rate of strongly discordant results is < 5%, “acceptable” if the rate of strongly discordant results is 50-10%, and “poor” if the rate is > 10%. As seen, there were no samples that rank as strongly discordant, and about 32% that rank as weakly discordant. Therefore, laboratory performance ranks as “good” and results for PLM-VE are judged to be acceptable.

#### Preparation Laboratory Quality Control Samples

**Preparation Blanks.** A preparation blank consists of asbestos free quartz sand and is processed with each batch of field soil/sediment samples. Preparation blanks determine if cross-contamination is occurring during sample preparation processes (i.e., drying, sieving, grinding, and splitting). A total of 9 (7%) preparation blanks were analyzed as part of the Phase I sampling program. Results for all preparation blanks were reported as non-detect by PLM-VE. These results indicate that preparation procedures utilized within the preparation laboratory did not introduce LA contamination.

**Preparation Splits.** Preparation splits are prepared by dividing a sample into two parts after drying but prior to sieving and grinding. Comparison of the preparation split results with the paired original field sample results helps to evaluate the variability that arises during preparation and analysis. As described above for field duplicate results, preparation splits are ranked as concordant if both the original sample results and the preparation split result report the same semi-quantitative classification. A total of 13 (10%) preparation splits were analyzed as part of the Phase I sampling program. As shown in Table 3 (Panel A), 12 (92%) of the original-split pairs analyzed by PLM-VE were in concordance. One of the preparation splits analyzed by PLM-VE was ranked as weakly concordant with the parent sample.

As shown in Table 3 (Panel B), 8 (73%) of the original-split pairs analyzed by the gravimetric method were in concordance. Three (27%) of the original-split pairs analyzed by the gravimetric method were ranked as weakly concordant. Since preparation splits may be authentically different due to within-sample heterogeneity, there are no acceptance criteria for preparation splits. The data suggest that results will generally be similar although differences due to small scale heterogeneity within samples may occur.

#### Analytical Laboratory Quality Control Samples

**Laboratory Duplicates.** A laboratory duplicate is a re-preparation of a soil sample slide by a different analyst than who performed the initial analysis. Laboratory duplicates are performed to evaluate potential analytical differences between analysts. As described above for field duplicate results, laboratory duplicate results are ranked as concordant if both the original sample result and the laboratory duplicate result report the same semi-quantitative classification. The acceptance criterion for laboratory duplicate analyses is that no more than 10% of all samples shall be discordant (assigned different PLM-VE bins). A total of 22 (16%) laboratory duplicates were analyzed as part of the Phase I sampling program. As shown in Table 4, all original-duplicate pairs were within concordance (100% assigned to the same PLM-VE bins). These results support the conclusion that the soil sample results for PLM-VE are reproducible and reliable and are not greatly influenced by differences in laboratory analysis techniques between analysts. However, this conclusion is limited by the fact that all samples identified for laboratory duplicate analysis were ranked as non-detect.

### **NON-ASBESTOS IN MINE WASTE**

#### Data Completeness

Results have been reported for all of the field samples as specified in the Phase I SAP (USEPA 2007). The minimum requirements were met for field QC samples and laboratory QC samples.

#### Data Validation

Full validation was conducted on all samples analyzed for non-asbestos analytes. All tables and figures generated for this SAP reflect corrected data.

### Field Quality Control Samples

**Trip Blanks.** A trip blank consists of analyte-free laboratory reagent water which accompanies the empty sample bottles to the field and is placed in each cooler containing samples scheduled for VOC analysis. The trip blank is used to indicate potential contamination by VOCs during sample shipping and handling. A total of 10 trip blanks were collected as part of the Phase I sampling program. Seven of these trip blanks could not be analyzed due to a mistake in the field whereby the sampler took pre-weighed methanol charged vials and filled them with water. Chloroform and acetone were detected in two trip blank samples. Acetone is listed as a common laboratory contaminant by USEPA (1989). In addition, acetone was not detected in any field samples, nor was chloroform; thus no action was taken based on these findings.

**Field Duplicates.** A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision. A total of 4 (11%) field duplicates were collected as part of the Phase I sampling program. Field duplicate results are compared to the original sample results based on the relative percent difference (RPD); the difference between two sample results divided by their mean and expressed as a percentage. The RPDs for analytes detected in both the original sample and its field duplicate were less than 35% (the apparent advisory limit in soil followed by the validators) for most analytes, with the exceptions noted in Table 5. No qualification is taken on field duplicate results.

**Performance Evaluation Samples.** PE samples for water and in soil are available through the EPA Quality Assurance Technical Support (QATS) program. A total of 3 soil PE samples containing a range of inorganic and organic analytes were evaluated as part of the Phase I sampling program. At this time, information on whether measured concentrations are within the PE acceptance criteria is not available. It is anticipated that these results will be provided as part of subsequent OU3 SAPs when they become available.

### Analytical Laboratory Quality Control Samples

The laboratory-based QC samples as described in the Phase I SAP (USEPA 2007) were analyzed to assess and document the quality of analytical results for non-asbestos parameters in surface water samples. All non-asbestos data were evaluated based on the following parameters:

- Data Completeness
- Holding Times
- Gas Chromatography/Mass Spectroscopy Instrument Tune

- Calibrations
- Blanks
- Surrogate Recovery
- Matrix Spike/Matrix Spike Duplicates
- Laboratory Control Samples
- Internal Standards (if applicable)
- Field Duplicates (if applicable)
- Compound Identification
- Compound Quantitation and Reporting Limits
- System Performance
- Other Laboratory QC Specified by the Method
- Overall Assessment of Data

Samples were qualified if quality control criteria were not met according to the following scheme:

**R:** Reported value is “rejected.” Resampling or reanalysis may be necessary to verify the presence or absence of the compound.

**J:** The associated numerical value is an estimated quantity because the quality control criteria were not met.

**U J:** The reported quantitation limit is estimated because quality control criteria were not met. Element or compound was not detected.

**N J:** Estimated value of a tentatively identified compound. (Identified with a CAS number.) ORGANICS analysis only.

**U:** The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.

**NR:** Result was not used from a particular sample analysis. This typically occurs when more than one result for a compound is reported due to dilutions and reanalyses.

In brief, 47 mine waste samples (38 field samples, 4 field duplicates, 3 PE samples, and 2 trip blanks) were qualified following validation as shown in Table 6. Results were qualified as “R” for some organophosphorus pesticides among three mine waste samples (2 field samples and 1 field duplicate) and two trip blanks. The organophosphorus pesticides were rejected on the basis of low recoveries for lab control samples (LCS). All

other analytes listed in Table 6 were qualified as estimated for not meeting certain QC criteria.

## **ASBESTOS IN FOREST SOILS**

### Data Completeness

Results have been reported for all of the field samples as specified in the Phase I SAP (USEPA 2007). The minimum requirements were met for field QC samples and laboratory QC samples. Note that the minimum number of laboratory QC samples as specified in the Phase I SAP (USEPA 2007) required for analysis is based on a minimum percentage across all media types.

### Data Validation

In accord with the Phase I SAP, asbestos data were validated by selection of 10% of the samples at random and performing a detailed comparison of the FSDS and the laboratory bench sheets to the data recorded in the EDDs. A list of any omissions or apparent errors were submitted to the field team in the case of the FSDS forms and to the analytical laboratory in regard to analytical results reported in the EDDs. These issues were addressed and corrected. All tables and figures generated for this report reflect corrected data.

### Field Quality Control Samples

**Field Duplicates.** A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision. Field duplicate results are ranked as concordant if both the original sample result and the field duplicate result report the same semi-quantitative classification. Results are ranked as weakly discordant if the original sample result and the field duplicate result differed by one semi-quantitative classification (e.g., Bin A vs. Bin B1). Results are ranked as strongly discordant if the original sample result and the field duplicate result differed by more than one semi-quantitative classification (e.g., Bin A vs. Bin B2). A total of 8 (11%) field duplicates were collected as part of the Phase I sampling program. All 8 field duplicates were analyzed by PLM-VE and 6 of these were also analyzed by gravimetric analysis. As shown in Table 7 (Panel A), seven of the original-duplicate pairs analyzed by PLM-VE were in concordance, and the other field duplicate was ranked as weakly discordant with the parent sample. This discordance may be due to analytical variability, but might also arise from authentic heterogeneity between the sediment samples. Similarly, as shown in Table 7 (Panel B), four of the original-duplicate pairs analyzed by gravimetric analysis are in concordance, and two are ranked as weakly discordant with the parent

sample. The data suggest that results will generally be similar although differences due to small scale heterogeneity in the samples may occur.

**Performance Evaluation Samples.** Performance Evaluation (PE) samples are samples of a matrix that contain a known and certified level of a contaminant. The results of PE sample analysis help evaluate analytical accuracy. PE samples for LA in soil are available from USGS. These PE samples were prepared by mixing uncontaminated soil samples from Libby with known amounts of LA collected from the mine, so the true mass fraction of LA is known. A total of 19 PE samples with LA levels spanning a wide nominal range were evaluated at the PLM analytical laboratory prior to analysis of the Phase I samples as part of the laboratory mentoring and training program for Libby to evaluate laboratory proficiency. As described above for field duplicates, PE samples are ranked as concordant if the PE sample results report the same semi-quantitative classification as the nominal asbestos concentrations. As shown in Table 2, 13 (68%) of the PE samples were in concordance with the nominal values. Six of the PE samples were ranked as weakly discordant with the nominal values. Consistent with Libby Laboratory Modification #73 (LB-000073), proficiency is ranked as “good” if the rate of strongly discordant results is < 5%, “acceptable” if the rate of strongly discordant results is 5-10%, and “poor” if the rate is > 10%. As seen, there were no samples that rank as strongly discordant, and about 32% that rank as weakly discordant. Therefore, laboratory performance ranks as “good” and results for PLM-VE are judged to be acceptable.

#### Preparation Laboratory Quality Control Samples

**Preparation Blanks.** A preparation blank consists of asbestos free quartz sand and is processed with each batch of field soil/sediment samples. Preparation blanks determine if cross-contamination is occurring during sample preparation processes (i.e., drying, sieving, grinding, and splitting). A total of 9 (7%) preparation blanks were analyzed as part of the Phase I sampling program. Results for all preparation blanks were reported as non-detect by PLM-VE. These results indicate that preparation procedures utilized within the preparation laboratory did not introduce LA contamination.

**Preparation Splits.** Preparation splits are prepared by dividing a sample into two parts after drying but prior to sieving and grinding. Comparison of the preparation split results with the paired original field sample results helps to evaluate the variability that arises during preparation and analysis. As described above for field duplicate results, preparation splits are ranked as concordant if both the original sample results and the preparation split result report the same semi-quantitative classification. A total of 13 (10%) preparation splits were analyzed as part of the Phase I sampling program. As shown in Table 3 (Panel A), 12 (92%) of the original-split pairs analyzed by PLM-VE were in concordance. One of the preparation splits analyzed by PLM-VE was ranked as weakly concordant with the parent sample.

As shown in Table 3 (Panel B), 8 (73%) of the original-split pairs analyzed by the gravimetric method were in concordance. Three (27%) of the original-split pairs analyzed by the gravimetric method were ranked as weakly concordant. Since preparation splits may be authentically different due to within-sample heterogeneity, there are no acceptance criteria for preparation splits. The data suggest that results will generally be similar although differences due to small scale heterogeneity within samples may occur.

#### Analytical Laboratory Quality Control Samples

**Laboratory Duplicates.** A laboratory duplicate is a re-preparation of a soil sample slide by a different analyst than who performed the initial analysis. Laboratory duplicates are performed to evaluate potential analytical differences between analysts. As described above for field duplicate results, laboratory duplicate results are ranked as concordant if both the original sample result and the laboratory duplicate result report the same semi-quantitative classification. The acceptance criterion for laboratory duplicate analyses is that no more than 10% of all samples shall be discordant (assigned different PLM-VE bins). A total of 22 (16%) laboratory duplicates were analyzed as part of the Phase I sampling program. As shown in Table 4, all original-duplicate pairs were within concordance (100% assigned to the same PLM-VE bins). These results support the conclusion that the soil sample results for PLM-VE are reproducible and reliable and are not greatly influenced by differences in laboratory analysis techniques between analysts. However, this conclusion is limited by the fact that all samples identified for laboratory duplicate analysis were ranked as non-detect.

### **ASBESTOS IN TREE BARK**

#### Data Completeness

Results have been reported for all of the field samples as specified in the Phase I SAP (USEPA 2007). The minimum requirements were met for field QC samples and laboratory QC samples. Note that the minimum number of laboratory QC samples as specified in the Phase I SAP (USEPA 2007) required for analysis is based on a minimum percentage across all media types.

#### Data Validation

In accord with the Phase I SAP, TEM data were validated by selection of 10% of the samples at random and performing a detailed comparison of the FSDS and the laboratory bench sheets to the data recorded in the EDDs. A list of any omissions or apparent errors were submitted to the field team in the case of the FSDS forms and to the analytical



laboratory in regard to analytical results reported in the EDDs. These issues were addressed and corrected. All tables and figures generated for this SAP reflect corrected data.

#### Field Quality Control Samples

**Field Duplicates.** A field duplicate is a field sample that is collected at the same place and time as an original field sample. Field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision. Field duplicates for tree bark samples were collected at a rate of about 1 field duplicate per 10 field samples in accordance with the frequency specified in the Phase I SAP, resulting in eight field duplicates (out of 74 field samples). Table 8 summarizes the results of the original and duplicate tree bark samples. The original and duplicate sample results were compared using the Poisson ratio test recommended by Nelson (1982). As shown in Table 8, there were statistically significant differences in concentration among 5 out of the 8 pairs of original and duplicate tree bark samples. These results indicate that there is substantial variability in the tree bark data.

#### Analytical Laboratory Quality Control Samples

Detailed data for laboratory QC samples are provided electronically in Attachment A.3.

**Laboratory Blanks.** A laboratory blank is an analysis of a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples. Laboratory blanks determine if cross-contamination is occurring during sample analysis. Three laboratory blanks were analyzed prior to proceeding with the tree bark analyses. One filtration blank was also analyzed. No asbestos structures were observed in any of the TEM blank samples. This demonstrates that filter contamination from laboratory sources is not expected to influence asbestos results for tree bark samples collected as part of the Libby OU3 Phase I sampling activities.

**Recounts.** Recount analyses are performed by re-analyzing the same grid openings as in the original analysis, either by the same microscopist (recount same, RS) or by a different microscopist (recount different, RD). Three metrics were evaluated to assess the degree of agreement (concordance) between the original analysis and the recount analysis:

- total number of countable asbestos structures observed
- mineral class designation (LA, OA and C)
- structure dimensions (length, width)

Specific concordance criteria are detailed in Libby laboratory modification LB-000029.

A total of 63 grid openings in three samples were re-examined. Of these, one or more asbestos structures were observed in either the original and/or the recount analysis in 13 of the grid openings. In these 13 grid openings, a total of 56 unique asbestos structures were observed.

Table 9 presents the results of the structure count comparison. As seen, there was concordance for 61 of 63 grid openings. However, two fibers were detected in the recount analysis for the sample collected from SL45-10, whereas no fibers were detected in the sample for the original analysis.

Table 10 presents the results of the mineral assignments and the structure dimensions. For the sample collected from SL15-10, when one or more asbestos fiber was detected in paired grid openings, there was no difference between the original and recount analyses based on structure-specific comparisons.

**Repreparations.** A repreparation is an analysis of a TEM grid that is prepared from a new aliquot of the same field sample as was used to prepare the original grid. A repreparation analysis was conducted on one tree bark sample (collected at SL45-05) as part of the Phase I investigation. Table 11 summarizes the results of both the original analysis and the repreparation analysis. As seen, the LA levels in the original and preparation samples were not statistically different from each.

## REFERENCES

Nelson, W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

USEPA. 1989. Risk Assessment Guidance for Superfund (RAGS). Volume I. Human Health Evaluation Manual (Part A).

U.S. Environmental Protection Agency (EPA). 2007. *Phase I Sampling and Analyses Plan for Operable Unit 3 Libby Asbestos Superfund Site*. Prepared for the USEPA Region 8, Denver, Colorado with technical assistance from Syracuse Research Corporation and NewFields Boulder LLC, Boulder, CO. September 26, 2007.

## ATTACHMENT A.2

**Table 1. Concordance of Mine Waste Field Duplicates**

**Panel A. Field Duplicated (PLM-VE)**

		Field Duplicates			
		Bin A	Bin B1	Bin B2	Bin C
Original Result	Bin A	0	0	0	0
	Bin B1	0	0	1	0
	Bin B2	0	0	3	0
	Bin C	0	0	0	0

Total Pairs                      4  
 Concordant                      3 (75%)  
 Weakly Discordant            1 (25%)  
 Strongly Discordant          0 (0%)

**Panel B. Field Duplicates (Gravimetric)**

		Field Duplicates		
		ND	Tr	C
Original Result	ND	0	0	0
	Tr	0	2	0
	C	0	1	1

Total Pairs                      4  
 Concordant                      3 (75%)  
 Weakly Discordant            1 (25%)  
 Strongly Discordant          0 (0%)

Concordant pairs are shaded in gray.

## ATTACHMENT A.2

**Table 2. Concordance of Laboratory PE Samples by PLM-VE**

		Laboratory Results			
		Bin A	Bin B1	Bin B2	Bin C
Nominal Result	Bin A	2	0	0	0
	Bin B1	0	0	4	0
	Bin B2	0	0	5	1
	Bin C	0	0	1	6

Total Pairs                      19  
 Concordant                      13 (68%)  
 Weakly Discordant            6 (32%)  
 Strongly Discordant          0 (0%)

Concordant pairs are shaded in gray.

## ATTACHMENT A.2

**Table 3. Concordance of Preparation Splits by  
PLM-VE or gravimetric analysis**

**Panel A: Prep Splits (PLM-VE)**

		Prep Split Results			
		Bin A	Bin B1	Bin B2	Bin C
Original Result	Bin A	5	0	0	0
	Bin B1	1	2	0	0
	Bin B2	0	0	4	0
	Bin C	0	0	0	1

Total Pairs                      13  
 Concordant                      12 (92%)  
 Weakly Discordant              1 (8%)  
 Strongly Discordant            0 (0%)

**Panel B: Prep Splits (Gravimetric)**

		Prep Split Results		
		ND	Tr	C
Original Result	ND	4	2	0
	Tr	0	3	0
	C	0	1	1

Total Pairs                      11  
 Concordant                      8 (73%)  
 Weakly Discordant              3 (27%)  
 Strongly Discordant            0 (0%)

Concordant pairs are shaded in gray.

ND = non-detect

Tr = trace

## ATTACHMENT A.2

**Table 4. Concordance of Laboratory Duplicates by PLM-VE**

		Lab Duplicate Results			
		Bin A	Bin B1	Bin B2	Bin C
Original Result	Bin A	22	0	0	0
	Bin B1	0	0	0	0
	Bin B2	0	0	0	0
	Bin C	0	0	0	0

Total Pairs                      22  
 Concordant                      22 (100%)  
 Weakly Discordant            0 (0%)  
 Strongly Discordant          0 (0%)

Concordant pairs are shaded in gray.

## ATTACHMENT A.2

**Table 5. Relative Percent Difference (RPD) Between Detected Analytes**

<b>Analyte</b>	<b>MS-28 Original</b>	<b>MS-28 Duplicate</b>	<b>RPD</b>
Total Extractable Hydrocarbons	26 mg/kg-dry	15 mg/kg-dry	54%
Total Purgeable Hydrocarbons	3.2 mg/kg-dry	Not Detected	200%
Organic Carbon	1.41 wt%	0.46 wt%	102%
Mercury	Not Detected	0.1 mg/kg-dry	200%

<b>Analyte</b>	<b>MS-11 Original</b>	<b>MS-11 Duplicate</b>	<b>RPD</b>
Zinc	63 mg/kg	37 mg/kg	52%
Total Extractable Hydrocarbons	13 mg/kg	35 mg/kg	92%

<b>Analyte</b>	<b>MS-3 Original</b>	<b>MS-3 Duplicate</b>	<b>RPD</b>
Arsenic	2 mg/kg-dry	3 mg/kg-dry	40%
Copper	21 mg/kg-dry	40 mg/kg-dry	62%
C11 to C22 Aromatics	27 mg/kg-dry	14 mg/kg-dry	63%
C19 to C36 Aliphatics	42 mg/kg-dry	26 mg/kg-dry	47%
Total Extractable Hydrocarbons (DRO)	115 mg/kg-dry	51 mg/kg-dry	77%
Total Extractable Hydrocarbons (TEH)	163 mg/kg-dry	108 mg/kg-dry	66%

RPD = Relative Percent Difference

## ATTACHMENT A.2

Table 6. Validation Qualification for Non-Asbestos Analytes in Mine Waste Sample

[illegible]



## ATTACHMENT A.2

Table 6 (Continued). Validation Qualification for Non-Asbestos Analytes in Mine Waste Samples

Station ID	Field QC Type	Analyte Group	Analyte	Qualifiers		Reason for Qualification
				Original	Validation	
MS-31	FS	Metals	Barium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Iron	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-32	FS	Metals	Barium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Iron	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-33	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-34	FS	Metals	Barium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Iron	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-35	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-36	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-37	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-38	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-4	FS	Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
		VOCs	Chloroethane	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Demeton-O,S	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Phorate	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Disulfoton	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Fenthion	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
	FD	Organophosphorus Pesticides	Bolstar (Sulprofos)	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
		VOCs	Chloroethane	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Demeton-O,S	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Phorate	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Disulfoton	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Fenthion	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Bolstar (Sulprofos)	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
MS-5	FS	Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
		VOCs	Chloroethane	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Demeton-O,S	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Phorate	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Disulfoton	U	R	Low recoveries in laboratory control samples; below 10%
		Organophosphorus Pesticides	Fenthion	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Bolstar (Sulprofos)	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
		Organophosphorus Pesticides	Bolstar (Sulprofos)	U	UJ	LCS recoveries were less than the laboratory QC limit, but greater than 10%
MS-6	FS	Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Manganese	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-7	FS	Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-8	FS	Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
MS-9	FS	Metals	Antimony	U	UJ	Outside criteria limits for MS/MSD recoveries
DC-1 (PE Samples)	PE	Metals	Antimony	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Barium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Chromium	v	J	Outside criteria limits for MS/MSD recoveries
		Metals	Iron	v	J	Serial dilution %detects were less than 10% or result was less than 50x the MDL
DC-3 (Trip Blanks)	TB	VOCs	1,2,4-Trichlorobenzene	U	UJ	Percent differences in the continuing calibration were >25%
		VOCs	1,2,4-Trichlorobenzene	U	UJ	Percent differences in the continuing calibration were >25%
		VOCs	1,4-Dioxane	U	R	Extremely low RRF in the initial calibration
		VOCs	1,4-Dioxane	U	R	Extremely low RRF in the initial calibration

U = non-detected

v = detected

J = estimated

R = rejected

FS = Field Sample

FD = Field Duplicate

PE = Performance Evaluation

TB = Trip Blank

MS/MSD = Matrix Spikes/Matrix Spike Duplicates

RRF = Relative Response Factor

MDL = Method Detection Limit

## ATTACHMENT A.2

**Table 7. Concordance of Forest Soil Field Duplicates**

**Panel A. Field Duplicates (PLM-VE)**

		Field Duplicates			
		Bin A	Bin B1	Bin B2	Bin C
Original Result	Bin A	7	1	0	0
	Bin B1	0	0	0	0
	Bin B2	0	0	0	0
	Bin C	0	0	0	0

Total Pairs                      8  
 Concordant                      7 (88%)  
 Weakly Discordant            1 (13%)  
 Strongly Discordant          0 (0%)

**Panel B. Field Duplicates (Gravimetric)**

		Field Duplicates		
		ND	Tr	C
Original Result	ND	4	2	0
	Tr	0	0	0
	C	0	0	0

Total Pairs                      6  
 Concordant                      4 (67%)  
 Weakly Discordant            2 (33%)  
 Strongly Discordant          0 (0%)

Concordant pairs are shaded in gray.

**Table 8. Comparison of Original-Duplicate Tree Bark Pairs**

StationID	Approximate Distance From Mine (miles)	ORIGINAL			FIELD DUPLICATE			Poisson Rate Comparison (95% CI)
		N LA	Sensitivity (1/cm <sup>2</sup> )	Total LA Loading (MS/cm <sup>2</sup> )	N LA	Sensitivity (1/cm <sup>2</sup> )	Total LA Loading (MS/cm <sup>2</sup> )	
SL45-05	2.5	8	5.1E+03	0.04	51	1.1E+04	0.57	Original loading rate is significantly less than duplicate.
SL15-06	3.0	53	3.1E+04	1.62	14	9.4E+03	0.13	Original loading rate is significantly greater than duplicate.
SL75-13	5.0	6	9.0E+03	0.05	1	9.0E+03	0.01	Loading rates are not different.
SL75-15	6.0	30	8.7E+03	0.26	31	7.6E+03	0.24	Loading rates are not different.
SL195-06	3.0	51	1.5E+04	0.78	21	8.7E+03	0.18	Original loading rate is significantly less than duplicate.
SL255-05	2.5	51	9.8E+03	0.50	53	2.0E+04	1.06	Original loading rate is significantly less than duplicate.
SL135-04	2.0	52	8.1E+04	4.24	6	9.0E+03	0.05	Original loading rate is significantly greater than duplicate.
SL315-05	2.5	23	9.4E+03	0.22	11	9.4E+03	0.10	Loading rates are not different.

MS/cm<sup>2</sup> = million structures per square centimeter

LA = libby amphibole

Table 9. Concordance Results for Recount Analyses of Grid Openings

Analysis Summary				GO-Specific Evaluation		
Recount Type	Station ID	Grid	GO	LA Structure Count		
				Original	Recount	Concordant?
Recount Same	SL15-10	7	B3	0	0	Yes
		7	B5	0	0	Yes
		7	D1	0	0	Yes
		7	D3	0	0	Yes
		7	D7	0	0	Yes
		7	D9	0	0	Yes
		7	F1	0	0	Yes
		7	F3	0	0	Yes
		7	F5	0	0	Yes
		7	F8	0	0	Yes
		7	F10	0	0	Yes
		7	I2	0	0	Yes
		7	I4	0	0	Yes
		7	I6	0	0	Yes
		7	I8	0	0	Yes
		8	I9	0	0	Yes
		8	I7	0	0	Yes
		8	I5	0	0	Yes
		8	G4	0	0	Yes
		8	E2	1	1	Yes
		8	C3	0	0	Yes
		9	I6	1	1	Yes
		9	G3	1	1	Yes
		9	D3	0	0	Yes
		9	B6	0	0	Yes
		9	B9	0	0	Yes
Recount Different	SL45-10	1	J9	0	1	No
		1	J7	0	0	Yes
		1	J5	0	0	Yes
		1	J3	0	0	Yes
		1	J1	0	0	Yes
		2	J3	0	1	No
		2	J7	0	0	Yes
		2	J10	0	0	Yes
		2	F2	0	0	Yes
		2	G5	0	0	Yes
		2	E8	0	0	Yes
		2	C4	0	0	Yes
		2	C8	0	0	Yes
		2	A4	0	0	Yes
		2	A6	0	0	Yes
		3	J1	0	0	Yes
		3	J3	0	0	Yes
		3	J5	0	0	Yes
		3	J7	0	0	Yes
		3	J9	0	0	Yes
		3	H2	0	0	Yes
		3	H4	0	0	Yes
		3	H6	0	0	Yes
		3	H8	0	0	Yes
		3	H10	0	0	Yes
		3	F2	0	0	Yes
		3	F4	0	0	Yes
		3	F6	0	0	Yes
		3	F8	0	0	Yes
	SL195-06	1	E4	6	6	Yes
		1	E6	9	9	Yes
		1	E8	4	4	Yes
		1	E10	5	5	Yes
		1	B6	8	8	Yes
		2	C6	4	4	Yes
		2	C8	4	4	Yes
		2	C10	11	11	Yes

GO = grid opening

LA = libby amphibole

Table 10. Detailed Structure Concordance Results for Recount Analyses of Tree Bark Samples with One or More Structures Observed

Recount Type	Index ID	Grid	GO	ORIGINAL					RECOUNT					CONCORDANT?		
				Mineral Class	Struc Type	Length (um)	Width (um)	AR	Mineral Class	Struc Type	Length (um)	Width (um)	AR	Mineral Class	Length	Width
Recount Same	SL15-10	8	E2	LA	F	8.5	0.9	9.4	LA	F	8.5	1.0	8.5	Yes	Yes	Yes
		9	I6	LA	MF	2.5	0.1	25.0	LA	MF	2.5	0.2	12.5	Yes	Yes	Yes
		9	G3	LA	F	5.8	0.3	19.2	LA	F	5.8	0.4	14.4	Yes	Yes	Yes
Recount Different	SL195-06	1	E4	LA	F	7.3	0.3	24.3	LA	F	7.3	0.3	24.3	Yes	Yes	Yes
		1	E4	LA	F	6.0	0.8	7.5	LA	F	6.0	0.8	7.5	Yes	Yes	Yes
		1	E4	LA	F	1.7	0.25	6.8	LA	F	1.7	0.25	6.8	Yes	Yes	Yes
		1	E4	LA	MF	1.7	0.2	8.5	LA	MF	1.7	0.2	8.5	Yes	Yes	Yes
		1	E4	LA	MF	1.6	0.2	8.0	LA	MF	1.6	0.2	8.0	Yes	Yes	Yes
		1	E4	LA	F	1.2	0.25	4.8	LA	F	1.2	0.25	4.8	Yes	Yes	Yes
		1	E6	LA	F	7.0	0.7	10.0	LA	F	8.0	0.7	11.4	Yes	No	Yes
		1	E6	LA	F	3.8	0.3	12.7	LA	F	4.5	0.3	15.0	Yes	No	Yes
		1	E6	LA	F	2.6	0.25	10.4	LA	F	2.6	0.25	10.4	Yes	Yes	Yes
		1	E6	LA	F	15.9	0.6	26.5	LA	F	16.9	0.6	28.2	Yes	Yes	Yes
		1	E6	LA	F	14.4	2.2	6.5	LA	F	14.4	2.2	6.5	Yes	Yes	Yes
		1	E6	LA	B	2.3	0.4	5.8	LA	B	2.3	0.4	5.8	Yes	Yes	Yes
		1	E6	LA	F	2.9	0.45	6.4	LA	F	3.0	0.5	6.0	Yes	Yes	Yes
		1	E6	LA	F	1.8	0.25	7.2	LA	F	1.8	0.25	7.2	Yes	Yes	Yes
		1	E6	LA	F	4.2	0.8	5.3	LA	F	4.2	0.8	5.3	Yes	Yes	Yes
		1	E6	LA	F	7.0	0.4	17.5	LA	F	7.5	0.45	16.7	Yes	Yes	Yes
		1	E8	LA	F	10.0	0.45	22.2	LA	F	10.0	0.45	22.2	Yes	Yes	Yes
		1	E8	LA	F	3.8	0.5	7.6	LA	F	3.8	0.5	7.5	Yes	Yes	Yes
		1	E8	LA	F	3.5	0.6	5.8	LA	F	3.5	0.6	5.8	Yes	Yes	Yes
		1	E8	LA	MF	8.0	0.7	11.4	LA	MF	8.0	0.7	11.4	Yes	Yes	Yes
		1	E8	LA	F	3.1	0.5	6.2	LA	F	3.1	0.5	6.2	Yes	Yes	Yes
		1	E10	LA	F	11.0	0.3	36.7	LA	F	11.0	0.8	13.8	Yes	Yes	No
		1	E10	LA	F	30.0	0.8	37.5	LA	F	30.0	0.8	37.5	Yes	Yes	Yes
		1	E10	LA	F	2.3	0.25	9.2	LA	F	2.5	0.25	10.0	Yes	Yes	Yes
		1	E10	LA	F	5.0	0.25	20.0	LA	F	5.0	0.25	20.0	Yes	Yes	Yes
		1	E10	LA	F	1.0	0.2	5.0	LA	F	1.0	0.2	5.0	Yes	Yes	Yes
		1	B6	LA	MF	2.4	0.6	4.0	LA	MF	2.5	0.6	4.2	Yes	Yes	Yes
		1	B6	LA	MF	7.0	0.5	14.0	LA	MF	7.0	0.5	14.0	Yes	Yes	Yes
		1	B6	LA	F	2.9	0.2	14.5	LA	F	3.0	0.2	15.0	Yes	Yes	Yes
		1	B6	LA	F	2.8	0.1	28.0	LA	F	2.8	0.1	27.5	Yes	Yes	Yes
		1	B6	LA	F	1.2	0.15	8.0	LA	F	1.2	0.15	8.0	Yes	Yes	Yes
		1	B6	LA	F	29.0	1.1	26.4	LA	F	29.0	1.1	26.4	Yes	Yes	Yes
		1	B6	LA	F	5.2	0.8	6.5	LA	F	5.3	0.75	7.0	Yes	Yes	Yes
		1	B6	LA	F	6.9	0.6	11.5	LA	F	7.0	0.6	11.7	Yes	Yes	Yes
		2	C6	LA	MF	2.5	0.7	3.6	LA	MF	2.5	0.7	3.6	Yes	Yes	Yes
		2	C6	LA	F	7.7	1.1	7.0	LA	F	7.8	1.2	6.5	Yes	Yes	Yes
		2	C6	LA	MF	2.5	0.4	6.3	LA	MF	2.5	0.5	5.0	Yes	Yes	Yes
		2	C6	LA	F	0.9	0.15	6.0	LA	F	1.0	0.15	6.7	Yes	Yes	Yes
		2	C8	LA	MF	4.6	0.7	6.6	LA	MF	4.6	0.75	6.1	Yes	Yes	Yes
		2	C8	LA	MF	1.4	0.25	5.6	LA	MF	1.5	0.25	6.0	Yes	Yes	Yes
		2	C8	LA	F	2.8	0.25	11.2	LA	F	2.8	0.25	11.2	Yes	Yes	Yes
		2	C8	LA	F	3.2	0.5	6.4	LA	F	3.3	0.5	6.5	Yes	Yes	Yes
		2	C10	LA	F	5.6	0.3	18.7	LA	F	5.6	0.25	22.4	Yes	Yes	Yes
		2	C10	LA	F	4.0	0.7	5.7	LA	F	4.0	0.75	5.3	Yes	Yes	Yes
		2	C10	LA	F	2.5	0.25	10.0	LA	F	2.5	0.25	10.0	Yes	Yes	Yes
		2	C10	LA	F	0.9	0.15	6.0	LA	F	1.0	0.15	6.7	Yes	Yes	Yes
		2	C10	LA	MF	6.5	1	6.5	LA	MF	6.5	1	6.5	Yes	Yes	Yes
		2	C10	LA	F	2.7	0.15	18.0	LA	F	2.8	0.15	18.3	Yes	Yes	Yes
		2	C10	LA	F	1.0	0.15	6.7	LA	F	1.0	0.15	6.7	Yes	Yes	Yes
		2	C10	LA	F	6.5	0.4	16.3	LA	F	6.5	0.4	16.3	Yes	Yes	Yes
		2	C10	LA	F	1.1	0.25	4.4	LA	F	1.1	0.25	4.4	Yes	Yes	Yes
		2	C10	LA	F	2.5	0.25	10.0	LA	F	2.5	0.25	10.0	Yes	Yes	Yes
		2	C10	LA	F	4.8	0.6	8.0	LA	F	4.8	0.6	7.9	Yes	Yes	Yes

**Table 11. Repreparation Results by TEM for Tree Bark Samples**

Staion ID	ORIGINAL			REPREP			Poisson Rate Comparison (95% CI)
	N LA	Sensitivity (1/cm <sup>2</sup> )	Total LA (MS/cm <sup>2</sup> )	N LA	Sensitivity (1/cm <sup>2</sup> )	Total LA (MS/cm <sup>2</sup> )	
SL45-05	8	5.1E+03	0.04	7	8.7E+03	0.06	Loading rates are not different.

MS/cm<sup>2</sup> = million structures per square centimeter

LA = libby amphibole